ATTACHMENT II-1-1
ANALYTICAL PROCEDURES

MATRIX pH
METHOD EC-0700

1.0 SCOPE AND APPLICATION

1.1 Method EC-0700 is an electrometric procedure for measuring pH in liquids, soils and solids. The method is an adaptation of the method for Soil pH in SW-846 Method 9045.

2.0 SUMMARY OF METHOD

2.1 The soil sample is mixed with Type II or deionized water. The pH of the solution is then measured with a pH meter.

3.0 INTERFERENCES

3.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. Strong acid solutions, with a true pH of <1 may give incorrectly high pH measurements.

3.2 Temperature fluctuations will cause measurement errors.

3.3 Errors will occur when the electrodes become coated. If an electrode becomes coated with an material that will not rinse free (e.g. oily), the electrode can be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with water.

4.0 APPARATUS AND MATERIALS

4.1 pH Meter-temperature compensating
4.2 pH Electrode(s)
4.3 Beaker, 50-ml minimum
4.4 Magnetic stirrer
4.5 Magnetic stirring bar
4.6 Timing device (clock, watch, or timer)

5.0 REAGENTS

5.1 Water: ASTM Type II

5.2 Standard commercial pH solutions at or near pH of 4, 7, and 10.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 No preservatives are required for this method. The sample will be obtained by following Attachment II-1, *Waste Analysis Plan*.

7.0 PROCEDURE

The sample must be between 15^{o}C and 25^{o}C prior to analysis.

7.1 Calibration:

7.1.1 The pH meter will be calibrated with pH standards of 4 and 10. The equipment will be adjusted to within $\pm$ 0.3 pH units. The pH of 7 solution will be read and the equipment will be considered calibrated if this reading is also within $\pm$ 0.3 pH units.

7.1.2 These three standards will be checked every three hours of operation.

7.2 Sample Preparation: To 20 g of sample in a beaker, add 20 mL of Type II water. (More sample may be used as long as 1 mL of Type II water is added for every 1 g of sample.) Stir the suspension using a magnetic stirrer and a magnetic stirring bar. Allow the solution to stir for 5 minutes.

7.3 Measurement of pH: Immerse the electrodes deep enough to establish a good electrical contact and a stable reading. Read the pH meter.

8.0 DATA RECORDING AND MANIPULATION

8.1 Data Recording: Record the number on the meter in the laboratory analysis notebook.

8.2 Date and Initial Data. Place initials next to the number and ensure the date of analysis appears on the page.

8.3 Data Manipulation: No manipulation of the pH read from the meter is required. The value may be recorded directly.

9.0 QUALITY CONTROL

9.1 Electrodes must be thoroughly rinsed between samples. First, rinse the electrodes with laboratory tap water and check for signs of contaminants such as oil, grease or muds. If contaminants are still present, wipe electrode off with soft tissue cloth and repeat rinsing operations as necessary. When contaminants are removed from the electrode, rinse twice with ASTM Type II water.

9.2 The pH instrument will be checked against standards every 10 samples or three hours (whichever is more often) and recalibrated, if necessary, to ensure continuing calibration. The results of the check or recalibration will be recorded in the laboratory analysis notebook as well as the time that this step took place.

9.3 For pH results less than 2.0, the pH meter shall be checked against a standard which is less than or equal to 2.0. For pH results greater than 12.5 the pH meter shall be checked against a standard which is greater than or equal to 12.5. To ensure continuing calibration the result obtained must be within 0.05 of the actual value. If the result is not within 0.05 the meter shall be re-calibrated.

9.4 A duplicate will be run every 10 samples or three hours (whichever is more often) for a quality control. The results of the duplicate will be recorded in the laboratory analysis notebook as well as the time that this step took place.

10.0 SAFETY

10.1 Sampling and laboratory personnel must wear gloves, protective clothing, footwear, and laboratory eye wear with side guards or goggles when analyzing samples from incoming shipments.

11. SAMPLE DISPOSAL

11.1 Samples will be disposed of in accordance with Section VII.8 of Attachment II-1, *Waste Analysis Plan*.

ATTACHMENT II-1-1
ANALYTICAL PROCEDURES

PAINT FILTER LIQUIDS TEST
METHOD EC-0725

1.0 SCOPE AND APPLICATION

1.1 Method EC-0725 is used to determine the presence of free liquids in a sample of incoming waste. This method is an adaptation of the method for the Paint Filter Liquids Test in SW-846 Method 9095.The procedure of this method is equivalent to SW-846 Method 9095.

2.0 SUMMARY OF METHOD

2.1 A measured amount of a sample of waste is placed in a paint filter. If any liquid portion of the sample passes through and drops from the filter within the 5-minute test period, the material is considered to contain free liquids. (In some instances solid-phase soil, dirt, or dust particles may fall from the paint filter. This occurrence is expected with fine solids. No conclusion of free liquids will be made based on an observation of this occurrence.)

3.0 INTERFERENCES

3.1 Filter media were observed to separate from the filter cone on exposure to alkaline materials. This development causes no problem if the sample is not disturbed.

4.0 APPARATUS AND MATERIALS

4.1 Conical paint filter: Mesh number 60

4.2 Glass funnel: If the paint filter, with the waste, cannot sustain its weight on the ring stand, then a fluted glass funnel or a glass funnel with a mouth large enough to allow at least 1 in. of the filter mesh to protrude should be used to support the filter. The funnel is to be fluted or have a large open mouth in order to support the paint filter yet not interfere with the movement, to the graduated cylinder, of the liquid that passes through the filter mesh.

4.3 Ring stand and ring, or tripod.

4.4 Graduated Cylinder or Beaker.

4.5 Timing device (clock, watch, or timer)

5.0 REAGENTS

5.1 None

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 The sample will be obtained by following Attachment II-1, *Waste Analysis Plan*.

6.2 A 100-mL or 100-g representative sample is required for the test.

7.0 PROCEDURE

7.1 Place the ring on the ring stand, the paint filter on the ring, and the graduated cylinder or beaker beneath the narrow end of the cone filter so that any liquid drops that form may be collected in the graduated cylinder or beaker beneath the filter.

7.2 Place a 100-g or 100-mL sample in the filter. (A funnel may be used to support the filter.)

7.3 Allow sample to drain for 5 minutes into the graduated cylinder or beaker.

7.4 If any liquid from the sample collects in the graduated cylinder or beaker in the 5-minute period, then the material is deemed to contain free liquids.

8.0 DATA RECORDING AND MANIPULATION

8.1 If there are no liquids collected in the graduated cylinder, then record A "Pass" in the laboratory analysis notebook.

8.2 If there are liquids present in the graduated cylinder, then record "Fail".

8.3 Optional: Record observations as to whether there were any solid fines or minutia in the graduated cylinder at the conclusion of the test. (Note: A sample may still pass when there are only solids in the graduated cylinder at the conclusion of the procedure.)

8.4 Date and Initial Data. Place initials next to the notations of results in the laboratory analysis notebook. Ensure that the date of analysis appears on the page.

9.0 QUALITY CONTROL

9.1 Duplicate samples should be analyzed once each operating day. The results of this analysis must be recorded in the laboratory analysis notebook. (An operating day is a day that the test is run for an incoming shipment of waste.)

10.0 SAFETY

10.1 Sampling and laboratory personnel must wear gloves, protective clothing, footwear, and laboratory eye wear with side guards or goggles when sampling and analyzing incoming shipments.

11. SAMPLE DISPOSAL

11.1 Samples will be managed and disposed of in accordance with Section VII.8 of Attachment II-1, *Waste Analysis Plan*.

ATTACHMENT II-1-1
ANALYTICAL PROCEDURES

OXIDIZER/REDUCER TEST
METHOD EC-0750

1.0 SCOPE AND APPLICATION

1.1 Method EC-0750 is used to provide Envirocare with prompt qualitative information of the nature of a waste with respect to its electrochemical oxidizing and reducing properties.

2.0 SUMMARY OF METHOD

2.1 A measured amount of an acidified sample of waste is mixed with KI/starch solution. If a dark color is observed (brown, blue or violet) then the waste is considered to be oxidizing-type material. This is based on the reaction of I^- ions losing an electron and forming I_2 molecules in the reversible electrochemical oxidation reaction:

$$2\ I^- \text{--> } I_2 + 2e^-.$$

The equilibrium is shifted toward the iodine ion in the presence of a reducing agent such as sodium thiosulfate solution, $Na_2S_2O_3$ (aq) and, conversely may be shifted toward the iodine molecule in the presence of an oxidizing agent such as potassium nitrate KNO_3 or nitric acid.

4.0 APPARATUS AND MATERIALS

4.1 Scale for measuring a 20-g sample.
4.2 Two 400-mL beakers.
4.3 pH paper to indicate greater than or less than 7.
4.4 An eyedropper.
4.5 Filter paper.
4.6 Vacuum flask.

5.0 REAGENTS

5.1 10 percent solution of KI (aq).

5.2 10 percent solution of I_2 (aq).

5.3 3-5 percent solution of starch.

5.4 1:1 glacial acetic acid.

5.5 Potassium nitrate or nitric acid solution

5.6 Sodium thiosulfate or sodium sulfite solution.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 The sample will be obtained by following Attachment II-1, *Waste Analysis Plan*.

7.0 PROCEDURE

7.1 Place 20 g ("5g) of sample in the beaker.

7.2 Add 200 mL of distilled water to the sample.

7.3 Add 2 mL of 1:1 glacial acetic acid.

7.4 Check the solution with pH paper to ensure that it has a pH less than 7. If the solution is greater than 7, add 5 mL 1:1 glacial acetic acid and repeat this step until the pH is less than 7.

7.5 Using the paper filter (and vacuum pressure as needed), filter the sample, collecting the liquid phase in another beaker or flask. Add 20 mL of KI (aq) solution to the liquid phase in the beaker or flask.

7.6 Add an eyedropper of starch solution to the collected liquid.

7.7 Observe and record the color. If a dark color appears within 2 minutes, the waste is considered to be an oxidizer.

7.8 If no color is observed, add a few drops of I_2 solution. If the dark color from the I_2 in the resultant solution fades, the waste is considered to be a reducer. If the solution does not fade, then the waste is not considered to have properties of an oxidizer or a reducer.

8.0 DATA RECORDING AND MANIPULATION

8.1 Record the observations. If the color changes with the addition of KI (aq), then record "OXIDIZER" and record the color observed. If the color of the I_2 solution fades, then record "REDUCER" and describe any observations upon the fading of the I_2 solution. If the color does not change after the KI (aq) was added or after the I_2 was added then record "NEITHER" and note any observation. These notations should be made in the laboratory analysis notebook.

8.2 Date and Initial Data. Place initials next to the notations of results in the laboratory analysis notebook. Ensure that the date of analysis appears on the page.

9.0 QUALITY CONTROL

9.1 Daily, samples of solutions of potassium nitrate or nitric acid and sodium sulfite or sodium thiosulfate should be run without adjusting pH to test for test results. The results of this procedure will be recorded in the laboratory analysis notebook.

9.2 Daily, a duplicate will be run for a quality control. The results of the duplicate will be recorded in the laboratory analysis notebook as well as the time.

10.0 SAFETY

10.1 Sampling and laboratory personnel must wear gloves, protective clothing, footwear, and laboratory eye wear with side guards or goggles when sampling and analyzing incoming shipments.

11. SAMPLE DISPOSAL

11.1 Samples will be managed and disposed of in accordance with Section VII.8 of Attachment II-1, *Waste Analysis Plan*.

ATTACHMENT II-1-1
ANALYTICAL PROCEDURES

CYANIDE/SULFIDE TEST
METHOD EC-0775

1.0 SCOPE AND APPLICATION

1.1 Method EC-0775 is used to provide Envirocare with prompt qualitative information of the nature of a waste, specifically, for the waste's composition with respect to the potential to generate cyanide or sulfide when exposed to an acidic environment.

1.2 The results from this method will indicate whether the sample releases a significant level of detectable quantities of either cyanide or sulfide and the relative level of vapor concentration.

2.0 SUMMARY OF METHOD

2.1 A sample of waste is acidified and the resulting acid vapors are tested for the presence of HCN and H_2S using Drager detector tubes.

2.2 CAUTION: This method and its quality control are to be performed in an operating vented hood.

3.0 CROSS-SENSITIVITY

3.1 Hydrogen sulfide.

3.1.1 2000 ppm SO_2 or 100 ppm NO_2 do not interfere with the reading.

(Source: instructions for use, May 1992).

3.2 Hydrogen cyanide.

3.2.1 100 ppm H_2S, 300 ppm NH_3, 200 ppm SO_2, 50 ppm NO_2, or 1000 ppm HCl do not interfere with the reading.

3.2.2 H_2S turns the pre-layer dark brown, but has no influence on the HCN reading.

3.2.3 Ammonia concentrations in excess of 300 ppm may decolor the reading again at the beginning of the layer.

3.2.4 Up to 1000 ppm acrylonitrile do not interfere with the reading.

3.2.5 HCN measurement is not possible in the presence of phosphine.

(Source: instructions for use, September, 1993)

4.0 APPARATUS AND MATERIALS

4.1 Drager Multi Gas Detector Pump (must be Model 21/31, Accuro, Accuro 2000, or Quantimeter 1000.)

4.2 Drager tube, hydrogen cyanide. (Range 2 to 30 ppm).

4.3 Drager tube, hydrogen sulfide. (Range 100 to 2,000 ppm).

4.4 Beaker, 100-ml.

4.5 pH paper, for detecting greater than or less than 2.

5.0 REAGENTS

5.1 Nitric acid, 1% by weight, minimum.

6.0 SAMPLE COLLECTION, PRESERVATION, PREPARATION, AND HANDLING

6.1 No preservatives are required for this method. The sample will be obtained by following Attachment II-1, *Waste Analysis Plan*.

6.2 The sample will be prepared by placing 10 grams of waste into the 100-ml beaker.

7.0 PROCEDURE

(Note to analyst: Prior to running this procedure, you may want to test a separate sample with a few drops of acid to see whether a reaction will result in two-phase flow. Based on these results, you may be able to better prepare for containment of the experiment.)

7.1 When using a new tube, break off both tips of the tube in the tube opener. When using a previously opened tube, remove the caps.

7.2 Insert the tube tightly in the pump. Arrow points toward the pump.

7.3 20 ml of acid is added to the sample beaker using all of the precautions associated with acid chemical handling.

7.4 Immediately upon adding the acid, the gas mixture above the sample is tested using the hydrogen sulfide or the hydrogen cyanide Drager gas detector tube. The required number of strokes is; one stroke for H_2S and five strokes for the HCN.

7.5 After the measurement period of 30 seconds for H_2S and 60 seconds for HCN, check for discoloration within the tube. Record the results of this check as outlined in section 8.0.

7.6 Observe possible cross sensitivities.

7.7 Flush the pump with air after operation.

7.8 The pH of the mixture is taken with paper to ensure that the mixture had a pH less than 2. If the pH was greater than 2, this procedure will be repeated using a 10 g sample and an additional 20 ml of acid.

7.9 Place another 10 grams of the waste into another 100-ml beaker. Repeat steps 7.1-7.8 for the remaining hydrogen sulfide or hydrogen cyanide Drager gas detector tube.

7.10 To reduce the amount of waste generated as a result of this procedure, the tests may be performed simultaneously on the same sample using two separate pumps.

8.0 DATA RECORDING AND MANIPULATION

8.1 Record the observations for hydrogen cyanide. If hydrogen cyanide is read to be detectable on the Drager tube after the procedure (indicating layer turns RED), then write "CYANIDE" in the laboratory analysis notebook. Record also the concentration indicated. If hydrogen cyanide is not read to be detected on the Drager tube after the experiment write "HCN ND" in the laboratory analysis notebook.

8.2 Record the observations for hydrogen sulfide. If hydrogen sulfide is read to be detectable on the Drager tube after the procedure (indicating layer turns BROWN), then write "SULFIDE" in the laboratory analysis notebook. Record also the concentration indicated. If hydrogen sulfide is not read to be detected on the Drager tube after the experiment write "H_2S ND" in the laboratory analysis notebook.

8.3 Date and Initial Data. Place initials next to the notations of results in the laboratory analysis notebook. Ensure that the date of analysis appears on the page.

9.0 QUALITY CONTROL

9.1 The tubes are to be stored away from direct sunlight.

9.2 The tubes are to be used by the expiration date.

9.3 Once per week, the bellows is checked for leaks by testing it with an unbroken detector tube and observing any leaks in the pressure. Leaks must be repaired before pump may be used again for fingerprint analysis.

9.4 Once per week, the bellows is checked for suction by allowing it to open after pressure is applied. Faulty suction must be repaired before pump may be used again for fingerprint analysis.

9.5 A duplicate will be run each day for a quality control. The results of the duplicate will be recorded in the laboratory analysis notebook as well as the time.

9.6 Drager tubes may be re-used for up to 10 samples provided that no indication of detection or interference is observed. The tubes shall be capped when not in use.

9.7 New Drager tubes shall be used for pre-shipment analysis.

10.0 SAFETY

10.1 CAUTION: Acid is corrosive to skin and eye tissue. Sampling and laboratory personnel must wear gloves, protective clothing, footwear, and laboratory eye wear with side guards or goggles when sampling or analyzing incoming shipments.

10.2 Avoid breathing fumes from the acid or the reaction of the acid with the sample. This method must be performed in an operating fume hood.

11. SAMPLE DISPOSAL

11.1 Samples and materials will be managed and disposed of in accordance with Section VII.8 of Attachment II-1, *Waste Analysis Plan*.

12.0 REFERENCE. Drager Detector Tube Handbook, 6th updated edition, 1985, Drager Multigas Detector Instructions for Use; H_2S - January 1990, May 1992 and HCN - September 1993.

ATTACHMENT II-1-1
ANALYTICAL PROCEDURES

PHOTOIONIZER "SNIFFER" TEST
METHOD EC-0800

1.0 SCOPE AND APPLICATION

1.1 Method EC-0800 is used to provide Envirocare with prompt information of the nature of a waste with respect to the waste's composition of volatile organic compounds.

1.2 This method will provide results which reflect the quantitative level of volatile organic chemicals present in vapors above the waste.

2.0 SUMMARY OF METHOD

2.1 A sample is collected in an air-tight container. When the sample is collected, the sample container must be sealed immediately upon placement of the waste into the container. Similarly, the sample must be analyzed for this method at the earliest opportunity upon arrival in the laboratory.

2.2 An opening is made in the sample container, and the sniffer probe is inserted into the container. The sniffer is read and the result is recorded.

3.0 INTERFERENCES

3.1 Hazardous as well as non-hazardous constituents may be detected by the photoionizer. However, non-listed volatile organic compounds may contribute to the waste's ignitability.

3.2 The sniffer results are somewhat a function of the distance of the probe from the sample and of the state of equilibrium in the container. Care should be taken to provide a situation of equilibrium within the sample container during the analysis.

4.0 APPARATUS AND MATERIALS

4.1 HNU or other brand photoionization unit with detector probe or port.
4.2 Standard sample of n-hexane or calibration span-gas.

5.0 REAGENTS

None.

6.0 SAMPLE COLLECTION, PRESERVATION, PREPARATION, AND HANDLING

None.

7.0 PROCEDURE

7.1 Standardize the photoionizer with n-hexane, calibration span-gas or some other standard chemical before use each day. Follow the manufacturer's operating procedures for standardization.

7.2 Take the container of waste and provide a place to insert the sniffer probe into the environment within the container. Insert the probe into the sample container. Use care to create a situation of equilibrium and as steady an analytical readout as possible within the sample container during the analysis.

8.0 DATA RECORDING AND MANIPULATION

8.1 Record the value read on the meter and the setting of the multiplier.
Note: values that are less than 10 ppm shall be recorded as <10

8.2 Multiply these values together and record the result.

8.3 Date and Initial Data. Place initials next to the notations of results in the laboratory analysis notebook. Ensure that the date of analysis appears on the page.

9.0 QUALITY CONTROL

9.1 Standardize the meter daily using n-hexane or another standard chemical. Make a notation as to the results of the standardization.

9.2 A duplicate will be run once each operating day for a quality control. The results of the duplicate will be recorded in the laboratory analysis notebook as well as the time.

9.3 For control of the container, whenever a container is used which is constructed of a material other than glass, a blank reading of the container must be performed within 12 hours prior to the container's use for sample collection. The results of the blank container analysis must be recorded in the laboratory analysis notebook. Containers which have results above two standard deviations of the mean blank measurement will not be used.

10.0 SAFETY

10.1 Sampling and laboratory personnel must wear gloves, protective clothing, footwear, and laboratory eye wear with side guards or goggles when sampling or analyzing incoming shipments.

11. SAMPLE DISPOSAL

11.1 Samples will be managed and disposed of in accordance with Section VII.8 of Attachment II-1, *Waste Analysis Plan*.

END OF ATTACHMENT II-1-1